



## Dynamic Nature of the Ligustilide Complex

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Monomeric phthalides such as *Z*-ligustilide (**1**) and *Z*-butylidenephthalide (**2**) are major constituents of medicinal plants of the Apiaceae family. While **1** has been associated with a variety of observed biological effects, it is also known for its instability and rapid chemical degradation. For the purpose of isolating pure **1** and **2**, a gentle and rapid two-step countercurrent isolation procedure was developed. From a supercritical CO<sub>2</sub> fluid extract of *Angelica sinensis* roots, the phthalides were isolated with high GC-MS purities of 99.4% for **1** and 98.9% for **2** and consistently lower qHNMR purities of 98.1% and 96.4%, respectively. Taking advantage of molarity-based qHNMR methodology, a time-resolved study of the dynamic changes and residual complexity of pure **1** was conducted. GC-MS and (qH)NMR analysis of artificially degraded **1** provided evidence for the phthalide degradation pathways and optimized storing conditions. Parallel qHNMR analysis led to the recognition of variations in time- and process-dependent sample purity and has impact on the overall assessment of time-dependent changes in complex natural products systems. The study underscores the importance of independent quantitative monitoring as a prerequisite for the biological evaluation of labile natural products such as monomeric phthalides.

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